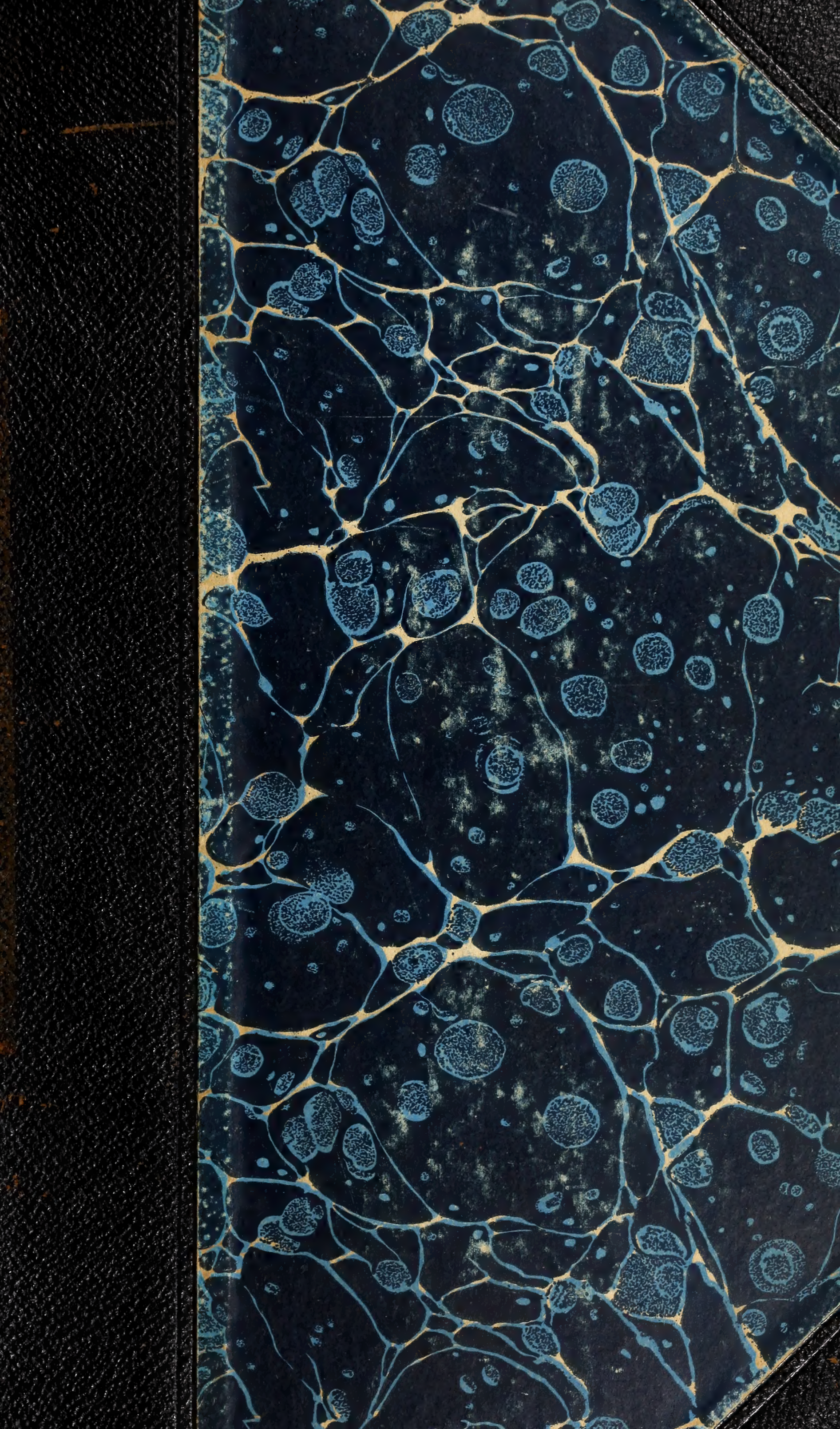

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THE PREPARATION OF AN EDIBLE OIL FROM CRUDE CORN OIL.

By A. F. Sievers, *Chemical Biologist*, and J. H. Shrader, formerly *Chemical Technologist, Drug, Poisonous, and Oil Plant Investigations.*

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SCOPE OF THE INVESTIGATIONS.

In a study made in 1919 of the production of corn oil in the United States,[1] in the course of which special attention was given to the manufacture of corn products as practiced in the so-called hominy mills and in starch and glucose plants, the fact developed that although there were 22 plants in the country which produced corn oil, only four of these were equipped at that time for producing refined edible oil from the crude product. The four plants refined not only the oil produced in their own mills but also a large proportion of that produced by other operators. There is now, however, a tendency among the smaller operators to consider the feasibility of refining their own crude oil, and consequently interest has been manifested in the refining process.

As a general rule, vegetable oils are refined[2] by a process consisting of three main operations: (1) Neutralizing the free fatty acids and removing the albuminous and coloring matter by treating the oil with caustic;[3] (2) bleaching the oil by heating with fuller's earth or decolorizing carbons, or both; and (3) deodorizing the oil by treating it with steam under reduced pressure at a high temperature, which also effects a further bleaching.

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[1] Sievers, A. F. The production and utilization of corn oil in the United States. U. S. Dept. Agr. Bul. 904, 23 p., 11 fig. 1920.

[2] The term "refining" as used in this bulletin refers to the complete process of purification of the oil and not to the alkali treatment only, in which sense it is frequently used.

This general procedure is followed, in the main, by most refiners of corn oil, but considerable difference of opinion exists regarding the technical details of the process. Consequently, it was thought advisable to make a thorough study of the preparation of an edible oil from the crude product with a view to clarifying some of the details in dispute.

This bulletin includes a discussion of experiments on the refining of corn oil under various modifications of the usual methods, a comparison of the economics of the methods proposed, and detailed plans of a commercial refining plant.

SMALL-QUANTITY EXPERIMENTS ON METHODS OF REFINING CORN OIL.

TREATMENT WITH CAUSTIC.

The technique involved in treating an oil with alkali varies according to the character of the oil and the individual opinion and experience of the operator. Oil obtained from the average quality of corn germs does not as a rule contain much more than 2 per cent of free fatty acids, and in many cases its acidity is as low as 1 per cent. If a good grade of corn is used the oil produced varies but slightly during the season with regard to acidity and color. It is possible, therefore, to refine many batches of such an oil with only slight, if any, changes in the process.

The oil used for the experimental work herein described was produced in a hominy mill and contained 1.76 per cent of free fatty acids. Method 1 was the so-called official method[4] used in control laboratories for grading cottonseed oils except that the maximum temperature was 55° C. instead of 45° C. In this method the quantity of caustic solution added is equal to 10 per cent of the weight of the oil. In method 2 only sufficient caustic solution is added to neutralize the free fatty acids present plus an additional quantity to remove the color and extraneous matter, the excess quantity being determined by the behavior of the oil under preliminary treatment. The experiments recorded are designed to show the proper strength of caustic to use with these two methods and, in the case of method 2, the quantity of excess which should be used, the two methods differing only in the quantity of caustic added. The technique employed in each case follows:

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[3] The term "caustic" as used in this bulletin refers in all cases to sodium-hydroxid solution.

[4] Interstate Cottonseed Crushers' Association. Official methods adopted by the chemists' committee and rules committee. III. Crude cottonseed oil. *In* Cotton Oil Press, v. 3, no. 3, p. 117. 1919.

The oil is placed in a suitable container equipped with a mechanical stirrer and agitated constantly while the caustic solution is slowly added. After being stirred for five minutes, to insure a thorough mixing, the oil is gently heated to 55° C. over a 15-minute period. When the oil approaches this temperature it breaks.[5] The stirring is now stopped and the oil allowed to stand for three hours at a temperature of 50° to 55° C. After cooling, it is separated from the deposited soapstock. In the experiments in which small quantities were used in beakers, the refining losses were determined by pouring off the oil from the soapstock.

In the first experiments with method 1, caustic solutions of 10°, 12°, 14°, and 16° Bé. were used, while with method 2 the solutions used were of 5°, 10°, 14°, and 20° Bé. strength. In each case the test was made on 100 grams of oil. With method 2, 10 per cent of caustic in excess of the quantity necessary to neutralize the free fatty acids was used. The results are shown in Table 1.

TABLE 1.—*Results of treating corn oil with several concentrations of caustic in connection with methods 1 and 2.*

Method and number of experiment.	Strength of caustic used.	Refining loss.	Free fatty acids in treated oils.	Character of settling and condition of soapstock.
Method 1:	*° Bé.*	*Per cent.*	*Per cent.*	
1	16	8.47	0.033	Rapid settling; soapstock is light and flaky and occludes much oil.
2	14	5.7	.031	Soapstock very much like that in experiment 1, but appears to contain less oil.
3	12	5.0	.037	Soapstock forms a semiliquid layer which hardens to soft petrolatum consistency on cooling.
4	10	4.6	.047	
Method 2:				
5	20	27.5	.076	Soapstock very slimy and does not settle firmly.
6	14	11.7	.047	Settles more rapidly than in experiment 5; soapstock slides.
7	10	11.9	.026	
8	5	12.5	.056	Soapstock liquid when hot; does not harden entirely on cooling.

The soapstock obtained by the two methods differs greatly. That obtained by method 1 is in all cases semiliquid while warm, and on cooling hardens to a consistency like that of soft petrolatum. Such soapstock occludes very little oil, with attendant low refining losses. It appears that the weaker concentrations of caustic, such as 10° and 12° Bé., give better results on corn oil.

Method 2 results in very high refining losses, because the soapstock in practically all cases is very slimy and occludes much oil. Accordingly, the most suitable concentration to use is apparently a strength of 14° Bé. It is evident that with this method it is beneficial to add some material to harden the soapstock in order to reduce the refining losses.

The next series of experiments was for the purpose of determining the quantity of excess caustic that should be used in method 2. In all cases 1.5 per cent of soda ash was added after the break, in order to harden the soapstock. Caustic of a strength of 14° Bé. was used in 10, 50, 100, and 200 per cent excess. Table 2 shows the results of these tests.

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[5] The appearance given to oil when coagulations of soap occur during refining.

TABLE 2.—*Results of using 10, 50, 100, and 200 per cent excess of 14° Bé. caustic with method 2.*

Experiment.	Excess caustic used.	Quantity of soda ash.	Refining loss.	Free fatty acid after treatment.	Character of settling and condition of soapstock.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
No. 15....	10	1.5	9.0	0.051	Soapstock very soft and slimy.
No. 16....	50	1.5	8.2	.041	Soapstock slightly more compact than that in experiment 15, but slides in mass.
No. 17....	100	1.5	8.0	.033	Soapstock very much like that in experiment 16.
No. 18....	200	1.5	7.2	.070	Soapstock very much like that in experiments 15 and 16.

The foregoing tests show that although not enough soda ash was used to make the soapstock firm it resulted in the soapstock settling to a much thinner layer, so that much less oil was occluded. A number of experiments were performed in which other dehydrating substances were used; namely, anhydrous sodium sulphate, calcium sulphate (plaster of Paris), and common salt. Of these substances only the dry sodium sulphate compared favorably with the commercial soda ash in the hardening effect upon the soapstock. Table 3 shows the effect of using larger quantities of soda ash in connection with method 2.

TABLE 3.—*Effect of the use of 2 and 3 per cent of soda ash on the soapstock in connection with method 2 under several conditions.*

Experiment.	Strength of caustic used.	Excess caustic used.	Quantity of soda ash used.	Refining loss—		Condition of soapstock.
				By pouring off the oil.	After heating and recooling soapstock.	
	°*Bé.*	*Per cent.*	*Per cent.*			
No. 19........................	14	50	2	7.7	5.6	Firm.
No. 20........................	14	50	3	9.4	5.9	Do.
No. 21........................	14	100	2	6.7	5.4	Very firm.
No. 22........................	14	100	3	5.5	5.5	Do.
No. 23........................	14	200	2	7.3	5.9	Very firm and bulky.
No. 24........................	14	200	3	7.3	7.0	Do.

It will be seen from Table 3 that the refining loss can be very materially reduced by the use of 2 per cent of soda ash in connection with method 2, whether the excess caustic used is 50, 100, or 200 per cent. The use of an additional 1 per cent is of no advantage, because a greater bulk of soapstock results, with a consequent greater occlusion of oil. In all these tests the soapstock was firm and permitted the thorough draining off of the oil. It was found that when the soapstock was heated to about 50° C. and rehardened by cooling, more oil separated out and could be drained off. In this way the refining losses were still further reduced.

So far as refining loss is concerned, method 1 with 10° Bé. caustic and method 2 with 14° Bé. caustic, using 50 per cent excess and 2 per cent soda ash, give satisfactory results. The oil resulting from either method is low in free fatty acids, but that from method 1 appears to bleach somewhat better when treated with fuller's earth and deodorized, which is due no doubt to the greater excess of caustic used.

After being treated by method 1 the oil is quite turbid, because of a fine suspension of moisture and soap. That soap is present is evidenced by the fact that dehydration, while it clarifies the oil, leaves fine particles in suspension, which gradually settle out on cooling. Therefore, oils treated by this method must be washed in order to remove the soap. The oil from method 2, although somewhat turbid, evidently does not contain soap, because when the moisture is removed the oil is absolutely clear.

BLEACHING.

Vegetable oils are usually bleached by means of fuller's earth or finely divided carbon. Oils differ greatly in the ease with which they can be bleached, corn oil being particularly difficult. In order to formulate an exact method for bleaching this oil by means of fuller's earth, several experiments were made to establish (1) the proportion of earth necessary, (2) the proper temperature, and (3) the proper length of treatment.

In the first experiment four samples of a composite of caustic-treated oils were used with 2, 4, 7, and 10 per cent of standard fuller's earth,[6] respectively. One hundred grams of oil were heated, with constant stirring, to 110° C. and kept at that temperature about 10 minutes to remove all traces of moisture. This treatment is considered necessary, because the earth does not effect a satisfactory bleach if the oil contains any moisture. The temperature was held at 105° C., while the earth was added slowly and the stirring continued for five minutes. The oil was then rapidly filtered on a force filter. Figure 1 shows the extent to which the several proportions of earth removed the color. The readings in all cases were made in a 2-inch cell with a Lovibond tintometer. The quantity of oil available in these preliminary experiments was not sufficient to enable the use of the 5¼-inch cell, which is considered the standard.

The results show, as was to be expected, that, within the range of this experiment, the bleaching increases with the increase in the percentage of earth used. Seven per cent of earth appears to be the most beneficial, but from a practical standpoint 5 or 6 per cent is sufficient, since no reduction in red is obtained with higher proportions. There is a limit, of course, to the proportion of earth which may be used in practice. The expense involved would not justify the use of more than 6 per cent. The expense lies not only in the cost of the earth, but in the additional loss of oil which is entailed by the use of the greater quantities and the somewhat greater expense of filtering. Fuller's earth generally shows less bleaching efficiency when used in laboratory quantities than when used in large-scale operations. Hence, the use of a maximum of 5 or 6 per cent of earth, as generally practiced in refineries, would seem to be in accordance with the results of these tests.

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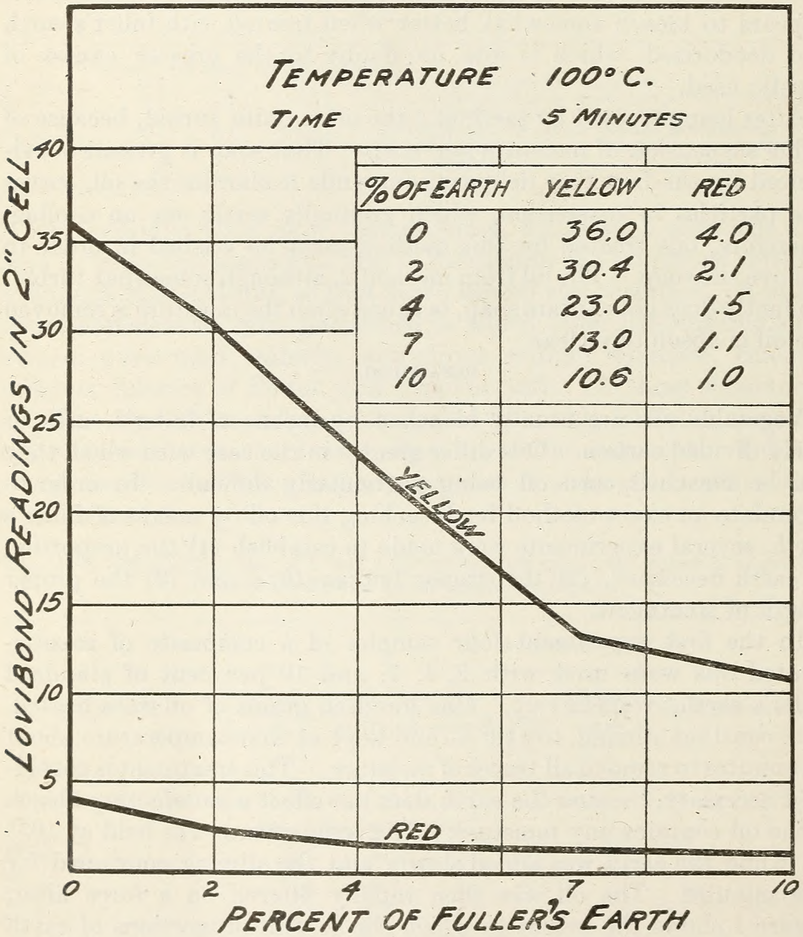
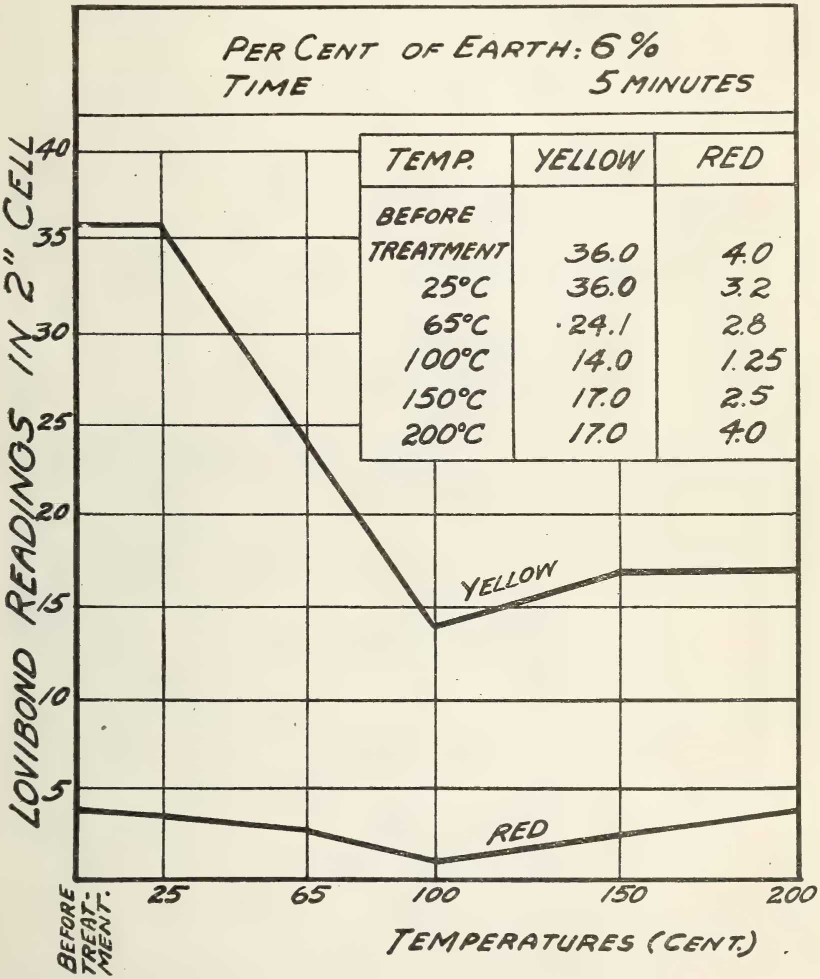
[6] Standard fuller's earth is recommended by the American Oil Chemists' Society for bleaching vegetable oils and may be obtained from the secretary of that society.



% OF EARTH	YELLOW	RED
0	36.0	4.0
2	30.4	2.1
4	23.0	1.5
7	13.0	1.1
10	10.6	1.0

FIG. 1.—Relation between the proportion of fuller's earth used and the extent of the bleaching effected.

Equally as important as the proportion of earth is the temperature; hence, experiments were made to determine the proper temperature range at which the oil should be treated. A sample of oil which had previously been dehydrated in the manner described was divided into five lots, each lot to be bleached with 6 per cent of fuller's earth at temperatures of 25°, 65°, 100°, 150°, and 200° C., respectively. In each case the oil was uniformly stirred and heated to the desired temperature, whereupon the earth was added slowly and the stirring continued for five minutes. The mixture was then filtered through a force filter. Figure 2 illustrates the results obtained.

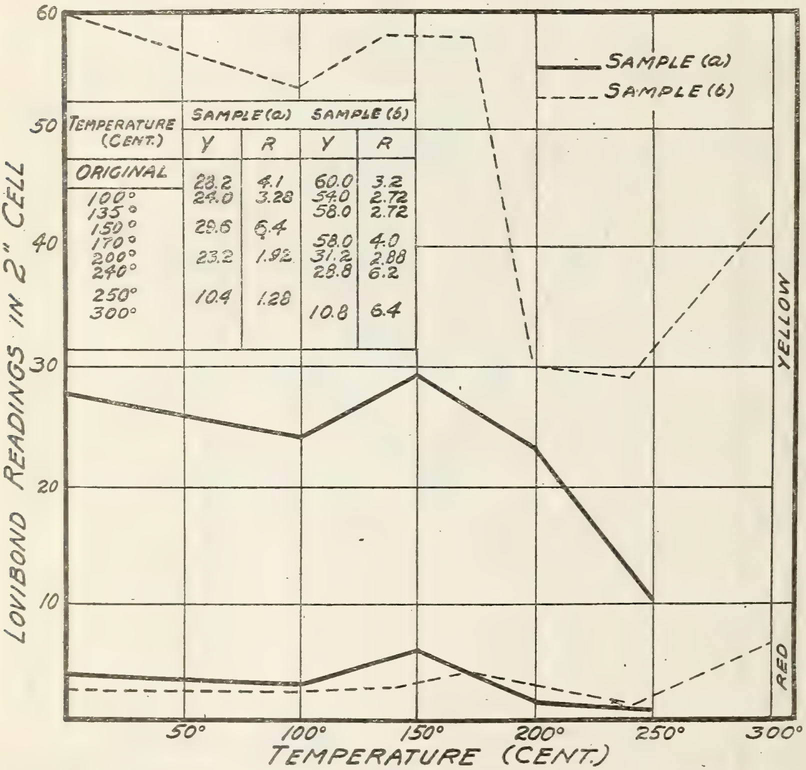


TEMP.	YELLOW	RED
BEFORE TREATMENT	36.0	4.0
25°C	36.0	3.2
65°C	24.1	2.8
100°C	14.0	1.25
150°C	17.0	2.5
200°C	17.0	4.0

FIG. 2.—Relation between the temperature maintained and the extent of the bleaching affected.

It is evident that the proper temperature at which to effect the treatment is about 100° C. At 25° C. practically no bleaching is obtained, while at temperatures considerably above 100° C. a less pronounced bleaching is effected. Since the oil must be dehydrated at a temperature somewhat above 100° C., treatment with fuller's earth at temperatures below that point, even if effective, would not be practicable, since time would be lost in waiting for the oil to cool.

In this connection, some tests were made to determine the bleaching effect of heating without the fuller's earth. Two 300-gram samples of oil, (*a*) of average color, and (*b*) of materially darker color, previously treated with caustic, were heated slowly, with constant stirring, to 250° C. At 100°, 150° and 200° C., respectively, the temperatures were maintained constant for five minutes; then samples for colorimetric readings were withdrawn and the heating continued. Figure 3 shows the extent to which the heating bleached these oils. A decided bleach seems to be effected at a temperature of 200° to 250° C. Above that temperature the oil apparently turns darker. Both samples of oil bleached slightly up to 100° C., then darkened somewhat until 150° C. was reached, and then bleached again as the temperature rose to 250° C. It is generally recognized

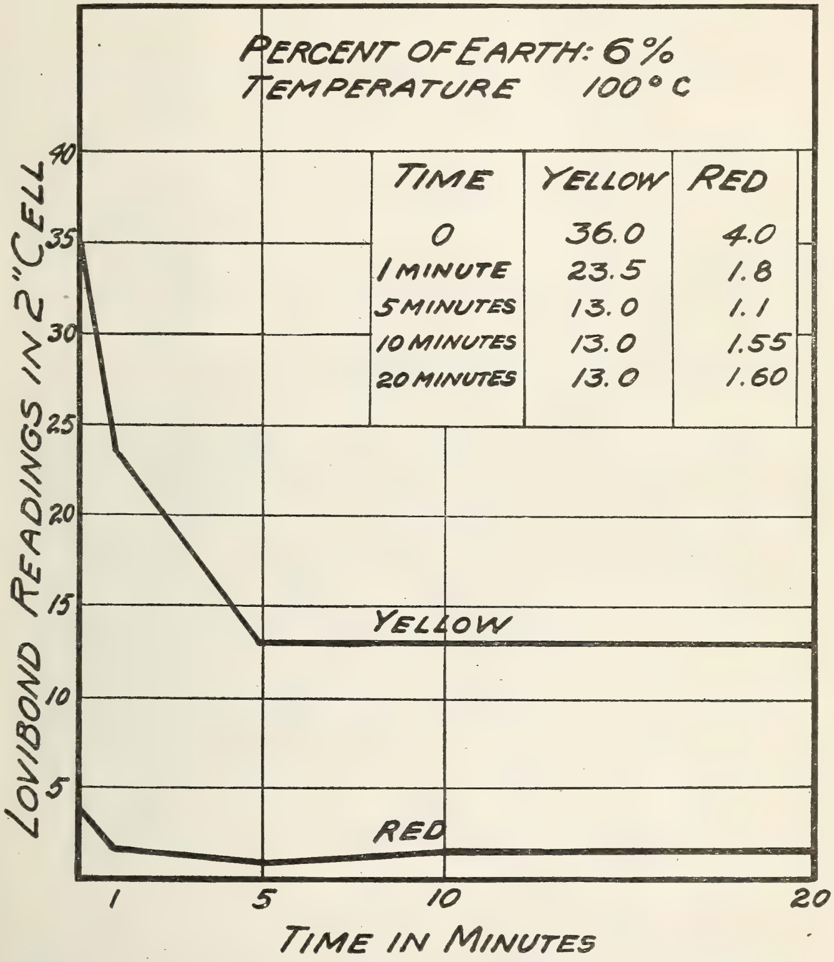


TEMPERATURE (CENT.)	SAMPLE (a)		SAMPLE (b)	
	Y	R	Y	R
ORIGINAL	28.2	4.1	60.0	3.2
100°	24.0	3.28	54.0	2.72
135°			58.0	2.72
150°	29.6	6.4		
170°			58.0	4.0
200°	23.2	1.92	31.2	2.88
240°			28.8	6.2
250°	10.4	1.28		
300°			10.8	6.4

FIG. 3.—The extent of bleaching effected in two samples of corn oil when heated to various temperatures up to 300° C.

that some vegetable oils will lighten in color on heating, but individual oils differ in this respect. Corn oil does not appear to bleach to a sufficient extent under this treatment alone.

The third experiment was designed to show the influence of the period of contact of the earth with the oil on the bleaching effected



TIME	YELLOW	RED
0	36.0	4.0
1 MINUTE	23.5	1.8
5 MINUTES	13.0	1.1
10 MINUTES	13.0	1.55
20 MINUTES	13.0	1.60

FIG. 4.—Relation between the time of contact with the fuller's earth and the bleaching effected.

Six per cent of standard fuller's earth was used at a temperature of 100° C. for periods of 1, 5, 10, and 20 minutes, respectively. From figure 4 it is evident that the maximum bleaching effect is obtained after a 5-minute contact and that about half of the bleaching is accomplished in the first minute.

Some difference of opinion exists as to whether fuller's earth effects a more efficient bleach if previously dehydrated. It is said to be the practice among some refiners to roast the earth, while others claim that the temperature used should be only high enough to remove external moisture and that excessive heating affects the porous character of the earth. Accordingly, three samples of oil were bleached with earth treated as follows: (1) No dehydration, (2) heated at 100° C. for 15 hours, and (3) roasted at high temperature for five minutes. Table 4 shows the results.

TABLE 4.—*Effect of the degrees of dehydration on the bleaching efficiency of standard fuller's earth.*

Treatment of the earth.	Color.[a]	
	Yellow.	Red.
No dehydration	[b] 13.0	1.1
Dehydrated at 100° C. for 15 hours [c]	21.5	1.1
Roasted at high temperature for five minutes	16.5	.9

[a] Colors read in 2-inch cell.
[b] Color of the oil before treating with earth: Yellow, 36.0; red, 4.0.
[c] This treatment resulted in a 12 per cent loss in moisture.

This experiment was repeated with almost identical results. Earth previously roasted effected a slightly better bleach on the red but somewhat poorer on the yellow, while earth dehydrated at 100° C. appears to be the least beneficial, for reasons not yet apparent. It appears, therefore, that better results are obtained on corn oil by using fuller's earth as it is found on the market.

Some oils can be more effectively bleached by means of a decolorizing carbon or a combination of such carbon with fuller's earth. Four of the principal commercial decolorizing carbons were tested on corn oil, alone and in combination with fuller's earth. The color of the oils thus treated was so manifestly the same that no readings were made, but the conclusion is warranted that the color in corn oil is not removed to any practicable degree by these carbons.

A further bleaching is effected during the deodorizing process.

DEODORIZING.

The process of deodorizing is the third and last principal step in the preparation of an edible oil. The trade demands an oil which is as nearly as possible without deleterious odor and taste, and it is mainly to remove such odor and taste that this last process is necessary. The treatment as followed at the present time has a further value, however, in that it frequently effects a considerable bleaching at the same time.

The method employed in the deodorization of oils consists essentially in blowing steam through the heated oil, usually under reduced pressure. This removes all volatile constituents and leaves a bland oil with practically no unpleasant odor or taste. The details of the process differ somewhat, according to the kind of oil that is being treated. In the case of corn oil the object of the deodorizing process is to remove the peculiar cereal flavor which is so characteristic of the oil.

The experiments performed in the deodorization of corn oil were conducted with a view of determining the proper temperature to be maintained and the time of treatment necessary to give the oil the best flavor and at the same time the lightest color. Some preliminary experiments consisted in blowing the previously neutralized and bleached oil with steam under atmospheric pressure. After four or five hours of such treatment the oil when separated from the condensed steam was found to be entirely odorless, but the color was not improved to any great extent. The oil thus treated was then divided into three lots, two of which were dehydrated and the other kept as a check. Table 5 gives the condition of the three oils after standing in well-filled bottles for some time.

TABLE 5.—*Effect of the presence of moisture on the keeping quality of deodorized corn oil.*

Sample.	Treatment for removing moisture.	Quality of oil after—		
		One week.	Four weeks.	Twelve weeks.
No. 1	None	Slightly rancid	Distinctly rancid and somewhat musty odor.	Very rancid and musty.
No. 2	Heated under vacuum to 95° C. for one hour.	Good quality	Good quality	Rancidity becoming noticeable.
No. 3	Stream of carbon dioxid passed through the oil for seven hours at 70° C.	do	do	Good quality.

It is evident that although steam at ordinary pressure volatilizes the odorous constituents of the corn oil and produces a very acceptable product as regards taste and odor, the oil must subsequently be dehydrated in order to prevent it from rapidly becoming rancid.

With very few exceptions vegetable oils are deodorized in commercial practice by blowing steam through the oil at a temperature of about 200° to 250° C. (392° to 437° F.) under high vacuum for about an hour, the time depending to some extent upon the character of the oil and the type of equipment. The object is to obtain an oil of satisfactory color and taste in the shortest time possible.

The oil is heated to 200° C. in the deodorizing tank either by direct heat or by means of superheated steam in closed coils placed in the bottom of the tank. Steam is then blown through the oil from a perforated coil. The practice differs with respect to this process, in some cases the steam being superheated. It is evident, however, that the use of superheated steam for this purpose will aid in keeping the oil at the desired temperature. After the oil is steamed for the necessary length of time the steam is turned off and the oil cooled under the reduced pressure to ordinary temperature.

In the experimental work on this process conducted in the laboratory, the oil was heated in a round-bottom glass flask on a sand bath by means of gas burners to 105° C. under a vacuum of 25 inches; the steam was then admitted and the heating continued to the desired temperature. All the experiments were conducted in a darkened room in order to avoid any possible bleaching effect of the light.

Two principal determinations were sought in this experimental work: (1) The proper temperature to be maintained and (2) the length of time necessary to give the best product. In determining the results obtained two qualities were considered: (1) The extent of the deodorization as indicated by the taste, odor, and acidity and (2) the bleaching effect obtained. In the first experiment samples of corn oil, previously neutralized and bleached with fuller's earth, were deodorized at 150°, 200°, and 300° C., respectively, the time of treatment varying from 15 to 120 minutes. The samples treated at 200° C. (392° F.) were uniformly of a very acceptable quality, even the one treated for only 15 minutes being entirely devoid of the peculiar cereal odor and taste of the untreated oil. The samples deodorized at 150° C. (302° F.) were practically the same in this respect. Those treated at 300° C. (572° F.) appeared to possess a slightly charred odor.

Table 6 shows the effect of the treatment on the percentage of free fatty acids.

TABLE 6.—*Percentage of free fatty acids in corn oil deodorized under various conditions.*

Temperature.	Percentage of free fatty acids [a] after treating for the number of minutes specified.						
	15	30	45	60	75	90	120
150° C	0.055	....	0.03	....	....	0.027	....
200° C	.07	0.046	.05	0.047	0.032	.023	0.024
300° C	.057	....	.045	....	....	.025	....

[a] The sample of corn oil used contained before the treatment 0.061 per cent of free fatty acids.

Before being deodorized by the foregoing method the oils gave a positive reaction for aldehydes by the Kreis test,[7] but after the deodorization the reaction was negative in all cases.

The treatment as described had a very marked bleaching effect. Figure 5 illustrates the extent to which bleaching was effected at the several temperatures.

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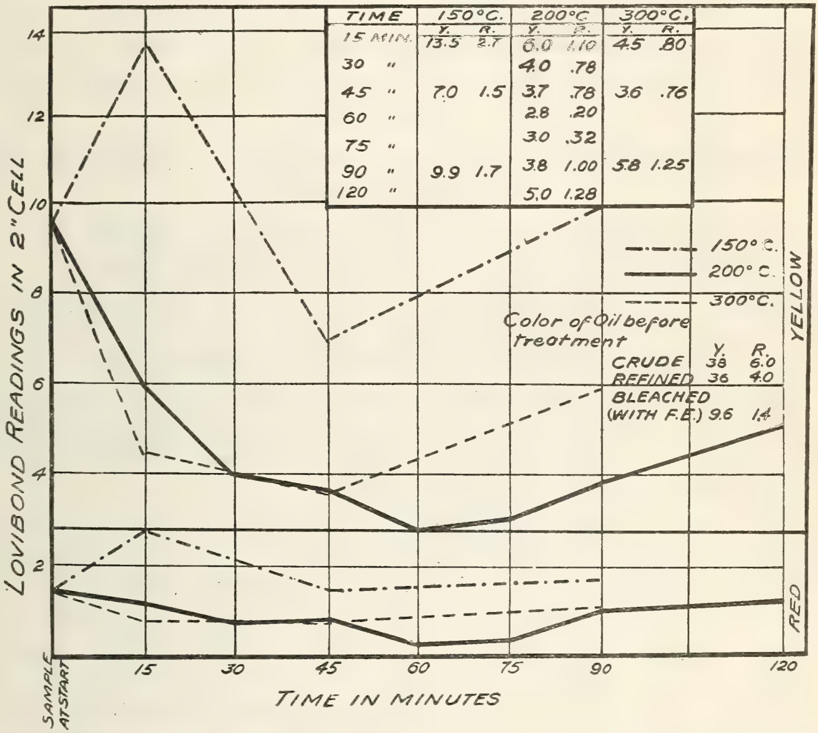
[7] Kerr, Robert H. Chemical tests for the detection of rancidity. *In* Jour. Indus. Engin. Chem., v. 10, no. 6, p. 471–475. 1918.

It is quite evident that the maximum bleach is obtained within the first hour of treatment. This is uniformly true at the three temperatures used in this experiment. At 150° C. the oil darkened during the first 15 minutes, increasing both in yellow and red, and then started to bleach, reaching its maximum in 45 minutes. At the higher temperatures the bleaching began at once and continued rapidly during the first 15 minutes, reaching its maximum in 60 minutes in the sample maintained at 200° C. and in 45 minutes in that at 300° C. The data included in figure 5 show that the bleaching effect obtained at 200° C. was greatest, resulting in a very light oil. The foregoing experiments justify the conclusion that corn oil can be effectively deodorized and bleached by treatment with saturated steam at 200° C. under a vacuum of 25 inches for one hour.



TIME	150°C.		200°C		300°C.	
	Y.	R.	Y.	R.	Y.	R.
15 MIN.	13.5	2.7	6.0	1.10	4.5	.80
30 "			4.0	.78		
45 "	7.0	1.5	3.7	.78	3.6	.76
60 "			2.8	.20		
75 "			3.0	.32		
90 "	9.9	1.7	3.8	1.00	5.8	1.25
120 "			5.0	1.28		

Color of Oil before treatment

	Y.	R.
CRUDE	38	6.0
REFINED	36	4.0
BLEACHED (WITH F.E.)	9.6	1.4

FIG. 5.—The extent of the bleaching effected by the deodorizing treatment continued for various periods and at various temperatures.

Since the bleaching obtained was so apparent, two samples of oil from the same source but not previously bleached with fuller's earth were deodorized at 200° C. for two and four hours, respectively. Table 7 shows the extent to which the oil was bleached and also a comparison of the results with those obtained from treating the oil with fuller's earth.

TABLE 7.—*Comparative bleaching effect of fuller's-earth treatment and the deodorizing process (200° C.) on corn oil, as indicated by Lovibond color readings in a 2-inch cell.*

Nature and time of treatment.	Color.	
	Yellow.	Red.
No treatment	60.0	4.8
Deodorization for two hours	10.4	3.2
Deodorization for four hours	11.2	3.6
Treatment with fuller's earth	19.2	4.0
Treatment with fuller's earth and subsequent deodorization for two hours at 200° C.	5.0	1.3

From these experiments it is evident that the deodorizing process results in a greater bleaching of the oil than the treatment with fuller's earth alone, but the finished oil is by no means as thoroughly bleached as when both treatments are employed.

COMPARATIVE REFINING QUALITIES OF CORN OIL FROM DRY AND WET PROCESS GERMS.

The manufacture of corn products falls into two general classes. In the manufacture of meal, flour, and hominy products it is necessary that the corn be degerminated in the dry condition; in the manufacture of the various starches and glucose products the corn is first thoroughly soaked before being degerminated.[8] This difference in the degerminating methods gives rise to two types of oil. The oil produced from the wet-process germs has a tendency to contain a somewhat higher percentage of free fatty acids. In other respects the two types are fairly similar.

To determine the relative quality of the edible oil obtainable from each of these two types of oil a sample of each was neutralized, bleached, and deodorized by the methods described. Sample A consisted of a composite mixture of oils from five large hominy mills, and sample B was a composite of oils from five starch and glucose plants. Of these crude samples oil A contained 0.92 per cent of free acids and oil B contained 2.17 per cent. Oil B was slightly darker than oil A. Both oils gave positive reactions to the Kreis reagent for aldehydes in dilutions of 1 to 20.

The oils were treated with caustic soda according to method 2, thus reducing the percentage content of acids to 0.019 in oil A and 0.023 per cent in oil B. A slight bleaching resulted from this treatment. The Kreis reagent showed a weak reaction for aldehydes in both oils at dilutions of 1 to 10. Both oils were then bleached with 6 per cent of fuller's earth, according to the method described. The treatment reduced the color in A to 10 yellow and 1.0 red and in B to 19.2 yellow and 3.0 red (2-inch cell).

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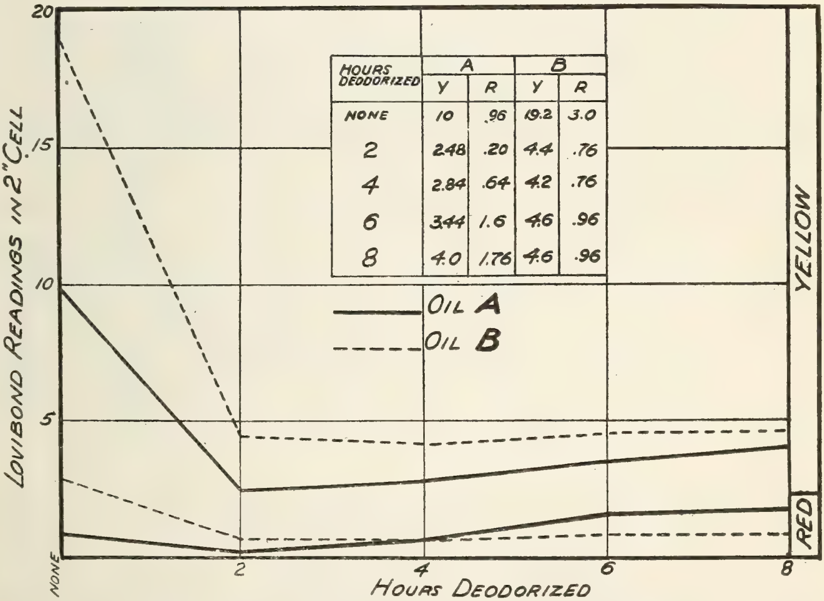
[8] Sievers, A. F. The production and utilization of corn oil in the United States. U. S. Dept. Agr. Bul. 904, 23 p. 11 figs. 1920.

The deodorization of these oils was extended over a long period, in order to determine just what effect such a protracted treatment would have. The oils were allowed to cool at 2-hour intervals, so that small samples might be removed to determine the color and free acidity. The process was then continued for another period, and so on. Table 8 shows the effect of the process of deodorization on the formation of free acids.

TABLE 8.—*Percentage of free fatty acids in corn oil from dry and wet process germs after various treatments.*

Sample.	Crude.	Alkali treated.	Deodorized—			
			Two hours.	Four hours.	Six hours.	Eight hours.
Oil A........................per cent..	0.92	0.019	0.024	0.02	0.042	0.047
Oil B..............................do....	2.17	.022	.032	.04	.051	.056

The diluted oils showed at all four stages of the deodorization treatment very slight reactions for aldehydes with the Kreis reagent.



HOURS DEODORIZED	A		B	
	Y	R	Y	R
NONE	10	.96	19.2	3.0
2	2.48	.20	4.4	.76
4	2.84	.64	4.2	.76
6	3.44	1.6	4.6	.96
8	4.0	1.76	4.6	.96

FIG. 6.—The comparative bleaching of oils A and B by the deodorizing treatment during successive 2-hour periods.

The marked effect of the deodorization treatment on the color of the oils is shown graphically in figure 6. The results obtained are further evidence that the bleaching is effected in the early stage of the process. Though oil B was considerably darker than oil A before they were deodorized, the finished oils after eight hours of treatment were very nearly the same color, B having less red but slightly more yellow than A. In fact, after two hours of treatment a comparison of the two oils leads to the conclusion that the color of the finished oil is not materially dependent upon the germs from which it is produced, whether from the dry or the wet process.

REFINING 10-POUND BATCHES OF CORN OIL TO DETERMINE THE COST OF THE CHEMICALS USED AND THE VALUE OF THE OIL LOST.

On the basis of conclusions drawn from the foregoing experiments on small quantities of oil, two batches of 10 pounds each were refined. The oil was obtained from a hominy plant and was therefore produced from dry-process germs. It contained 1.76 per cent of free fatty acids. One of the 10-pound batches was treated with caustic according to method 1, and the other batch according to method 2.

TREATMENT WITH CAUSTIC.

The 10-pound batch of oil to be treated by method 1 was placed in a 12-quart enameled bucket. A pound of 10° Bé. caustic was added and the mixture stirred for 30 minutes. Heat was then applied slowly with continued stirring over a period of 15 minutes until a temperature of 55° C. was reached. The break occurred as the temperature reached 45° C. The oil was warmed for three hours in a water bath at 55° C. and then allowed to cool to room temperature by standing overnight.

The oil, which will be designated C, was removed from the soapstock by suction through a rubber tube which could be moved about over the surface of the soapstock, a method similar to the commercial practice of pumping off the oil and skimming off the last portion by means of a swivel pipe. The soapstock was of a soft-petrolatum consistency, and the oil could all be drawn off with the exception of a thin layer. The refining loss was 6.05 per cent. Heating the soapstock did not result in separating out any more of the oil.

The neutralized oil was turbid, owing to the presence of moisture and soap. To remove the soap the oil was heated to 65° C. in the bucket, with constant stirring, and 5 per cent of a 10 per cent brine solution was added in a fine stream. Stirring was continued for 15 minutes and the oil then allowed to stand. The bulk of the brine settled out rapidly, but a turbidity remained in the oil even after it had stood all night. This turbidity was due to moisture rather than to soap, since heating completely clarified the oil and left no particles of soap in suspension, as was the case before the oil was washed.

The other 10-pound batch of oil was treated with 14° Bé. caustic according to method 2. The quantity required to neutralize the free fatty acids, plus 50 per cent excess, was added gradually and the mixture stirred for 30 minutes. After the temperature had risen gradually over a period of 15 minutes to 55° C. and the break had taken place, 2 per cent of soda ash was added and the stirring continued for five minutes. After warming for three hours and cooling overnight, the oil, which will be designated D, was drawn off in the manner described for oil C. The refining loss was 7.35 per cent. The soapstock was firm but contained considerable oil. After several holes had been cut in this soapstock with channels draining toward them, the bucket was placed on a steam bath for about 15 minutes. The holes quickly filled up with the oil, which was then drawn off. Several times these holes filled up, and by this means sufficient oil was recovered to reduce the refining loss to 5.44 per cent.

The neutralized oil, although somewhat turbid, contained no soap and consequently did not require washing. In this respect method 2 appears to have a distinct advantage over method 1.

BLEACHING.

Both oils were treated exactly alike from this point on. They were first dehydrated at 110° C., with constant stirring for 10 minutes. Five per cent of standard fuller's earth was then added and the stirring continued for 15 minutes with the temperature at about 105° C. The oils were then filtered rapidly through a Buchner funnel.

DEODORIZING.

Since there was at hand no apparatus large enough to deodorize the large quantities of oils C and D, smaller quantities of each were used. In each case the oil was treated at both 200° and 225° C. (392° and 437° F.) in glass flasks for one hour under a vacuum of 25 inches. A comparison of the finished oils is given in Table 9.

TABLE 9.—*Comparison of oils C and D after finished treatment.*

Oils and methods.	Color (5½-inch cell).								Free fatty acids after deodorizing at 200° C.	General quality of finished oils.
	After treatment with caustic.		After bleaching.		After deodorizing at—					
					200° C.		225° C.			
	Yellow.	Red.	Yellow.	Red.	Yellow.	Red.	Yellow.	Red.		
									Per cent.	
Oil C, method 1.	35	5.2	35	2.8	20	1.8	13	1.5	0.092	Odorless; no cereal taste; comparable to best commercial sample.
Oil D, method 2.	35	5.6	35	3.3	16	2.0	16	2.0	0.083	

ESTIMATED TOTAL COST OF REFINING CORN OIL.

The cost of refining vegetable oils includes such items as cost of chemicals, value of the oil lost in the several processes, fuel, labor, and overhead. In a relatively small refining operation, such as is here described, it is quite impossible to allocate the fuel, labor, and overhead expenses to the several operations. Accordingly, these items are charged in an estimated over-all cost of producing the finished edible oil. The cost of the chemicals used and the value of the oil lost are, however, highly specific to the several operations and can be charged thereto with a fair degree of accuracy.

TABLE 10.—*Cost of chemicals used and value of oil lost in treating 25,000 pounds of corn oil, containing 1.76 per cent of free fatty acids, by methods 1 and 2.*

Items of comparison.	Method 1.	Method 2.
Weight of oil......pounds..	25,000	25,000
Weight of free fatty acids (1.76 per cent)......do....	440	440
Sodium hydroxid necessary to neutralize the free fatty acids......do....	62.8	62.8
Total sodium hydroxid necessary......do....	[a] 163.7	94.2
Soda ash required......do....	....	500
Cost of caustic [b]......	$8.19	$4.71
Cost of soda ash [c]......	....	10.00
Value of oil lost in treatment, at 10 cents a pound:		
Method 1 (6.05 per cent)......	151.25	....
Method 2 (5.44 per cent)......	....	136.00
Total cost (chemicals and value of oil lost)......	159.44	150.71
Net cost (total cost, less the value of the soapstock, [d] $15.20 by method 1 and $17.32 by method 2)......	144.24	133.39
Net cost of chemicals used and value of oil lost per pound of crude oil......cent..	0.577	0.533
Net cost of chemicals used and value of oil lost per pound of refined oil......do...	0.628	0.564

[a] Method 1 requires 10 per cent of 25,000 pounds, or 2,500 pounds, of 10° Bé. caustic (6.55 per cent NaOH), equivalent to 163.7 pounds of sodium hydroxid.

[b] Caustic soda containing 76 per cent of sodium hydroxid is quoted at $3.75 per hundredweight, which is equivalent to 5 cents a pound of 100 per cent sodium hydroxid, delivered.

[c] Soda ash is quoted at $2 per hundredweight.

[d] Soapstock is valued according to its fatty acid content. The value of soapstock for methods 1 and 2 is derived as follows: Weight of soapstock—(1) Caustic (sodium hydroxid solution), method 1, 2,500 pounds; method 2, 1,000 pounds. (2) Soda ash, method 2, 500 pounds. (3) Oil lost, method 1, 1,512.5 pounds; method 2, 1,360 pounds. Total, method 1, 4,012.5 pounds; method 2, 2,860 pounds. The weight of fatty acids in the soapstock is calculated as follows: (1) Total weight of oil lost (oil and free acid), method 1, 1,512.5 pounds; method 2, 1,360 pounds. (2) Weight of free acids, method 1, 440 pounds; method 2, 440 pounds. (3) Weight of oil lost, method 1, 1,072.5 pounds; method 2, 920 pounds. (4) Weight of fatty acids in oil lost, method 1, 986.3 pounds; method 2, 846 pounds. (5) Total weight of fatty acids, (2)+(4), method 1, 1,426.3 pounds; method 2, 1,286 pounds. (6) Total weight of soapstock, method 1, 4,012.5 pounds; method 2, 2,860 pounds. (7) Fatty acids in soapstock, method 1, 35.5 per cent; method 2, 44.9 per cent. The money value of the soapstock is calculated by the following formula: $R \times P \times M \div 50 =$ value (dollars). In this formula R is the weight of the fatty acids (in pounds), P is the percentage of fatty acids in the soapstock, and M is the market price per pound (in dollars). If soapstock containing 50 per cent of fatty acids is quoted at $0.015 per pound, the value of the soapstock, according to this formula by method 1 is $1,426.3 \times 35.5 \times .015 \div 50 =$ \$15.20. By method 2 the value is $1,286 \times 44.9 \times .015 \div 50 =$ \$17.32.

CHEMICALS AND LOSS OF OIL.

It is evident that a very good quality of oil can be produced by both methods 1 and 2; hence, from this standpoint either method is practicable. The choice of the methods would appear, therefore, to depend upon which would be the more economical when such factors as loss of oil, cost of the chemicals used, and the time involved are considered. The cost of these items has been estimated on the basis of the results of refining the 10-pound batches for treating 25,000 pounds [9] of corn oil containing 1.76 per cent of free fatty acids, and a comparison of these estimates for both methods is presented in Table 10.

TREATMENT WITH CAUSTIC.

The difference in cost of chemicals and value of oil lost in the two methods is 0.064 cent per pound of refined oil in favor of method 2. This slightly lower cost of method 2 is due to the lower refining loss of oil and the slightly greater value of the soapstock, although the cost of the chemicals is higher than in method 1.

Since both methods were found to give a good quality of oil and since the difference in cost is not considerable, there would appear to be little, if any, advantage of one method over the other. As has been previously stated, however, method 2 does not require the washing of the oil with brine to remove the traces of soap, and this is a distinct advantage, especially from the standpoint of the time involved. On the other hand, this method calls for special handling of the soapstock in order to reduce the refining loss to the figure given in Table 10. In this respect method 1 has the advantage. It would appear, therefore, that the two methods are practically of equal value as to the ease of operation and cost.

BLEACHING.

In commercial practice it is generally assumed that the bleaching of vegetable oils with fuller's earth results in a loss of oil equivalent to 20 per cent of the weight of earth used. On this basis, the cost of the bleaching operation on the quantity of oil under discussion as regards the cost of earth and the value of the oil lost would be as shown in Table 11.

TABLE 11.—*Cost of material used and value of the oil lost in bleaching corn oil with fuller's earth.*

Items of comparison.	Method 1.	Method 2.
Weight of oil to be bleached..........pounds..	23, 487	23, 640
Quantity of fuller's earth required (5 per cent) [a]..........do....	1, 174. 3	1, 182
Loss of oil by treatment (20 per cent of weight of fuller's earth)..........do....	234. 8	236. 4
Cost of fuller's earth (2¼ cents a pound)..........	$26. 42	$26. 59
Cost of oil lost (10.5 cents a pound) [b]..........	24. 65	24. 82
Total..........	51. 07	51. 41
Cost of above items per pound of crude oil..........cent..	0. 204	0. 206
Cost of above items per pound of bleached oil..........do....	. 22	. 22

[a] This is a greater percentage of fuller's earth than is used for most vegetable oils, but commercial practice and laboratory tests appear to agree that such a quantity is necessary in the case of corn oil.

[b] This figure represents the cost of crude oil plus the approximate cost of the treatment with caustic.

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[9] The refining equipment described in this bulletin is designed to handle 50,000 pounds of oil a week. This quantity of oil would be produced in a hominy plant milling from 15,000 to 18,000 bushels of corn daily. In a starch plant, which produces at least twice as much oil from the same quantity of corn, the refinery would need to be proportionately larger.

The cost of the material used and the oil lost in the bleaching operation after treating the oil with caustic amounts to about 0.21 cent a pound of crude oil, of which approximately one half represents the cost of the fuller's earth and the other half the value of the oil lost in the operation.

DEODORIZING.

Deodorizing is an entirely mechanical operation and involves no charge for chemicals. There is a small loss of oil in the entrainment, which varies according to the local conditions, but even this small quantity of oil is recovered by means of the grease trap in the sewer.

FUEL.

The cost of fuel for caustic treatment and bleaching is comparatively low. For deodorization, however, the fuel charge is the main item of cost. Not only must the oil itself be heated to over 400° F., but it must be maintained at this temperature while approximately 25,000 pounds of steam are blown through it. Unless the steam has been previously superheated, it is evident that its passage through the oil will raise its temperature to that of the oil itself, which would operate to lower the temperature of the oil. Therefore not only must 25,000 pounds of steam be generated but it must be superheated to at least 450° F. before it can effectively serve its purpose. The oil itself must likewise be raised to this temperature, all of which accounts for the large quantity of fuel used. This consists not only of coal as burned under plant boilers for steam but also of fuel oil used in the direct-fired oil heater.

LABOR.

The labor necessary for handling the oil through the refinery is quite evenly distributed among the three principal operations. It is estimated that two men—one skilled workman and one unskilled laborer—can operate the plant under the general direction of the chemist and plant superintendent. A charge of $13.80 is made for these two workmen for each of the three operations, or a total of $41.40 for each batch of 25,000 pounds of oil. The cost of the technical supervision is charged to overhead by including the salaries of the chemist and plant superintendent with those of the general executive force and assuming that a tenth of their time will be devoted to matters connected with the refinery.

OVERHEAD.

The overhead item covers all plant charges not enumerated in the above, and consists of such costs as the following: Interest on a $40,000 equipment, building charge, water, insurance, power, office expenses, and selling charges.

Table 12 shows the distribution of costs for the several processes involved.

TABLE 12.—*Distribution of costs of refining 25,000 pounds of corn oil.*

Items.	Neutralizing.	Bleaching.	Deodorizing.	Total.
Chemicals	[a] $14. 71	$26. 59	....	$41. 30
Loss of oil	[b] 118. 68	24. 82	....	143. 50
Fuel	. 50	. 75	$12. 42	13. 67
Labor	13. 80	13. 80	13. 80	41. 40
Overhead	....	....	....	161. 19
Total	....	....	....	[c] 401. 06

[a] Caustic treatment according to method 2.
[b] This figure is obtained by subtracting the value of the soapstock from the value of the oil lost.
[c] Cost per pound, 1.6 cents.

DESCRIPTION OF THE EQUIPMENT AND THE METHOD OF HANDLING CORN OIL IN THE REFINERY.

The refinery illustrated in figure 7 is designed for installation in a corn-oil expelling plant. Since such a plant is generally provided with storage facilities for crude oil, the refining equipment as here designed provides for only one tank, *A*, for this purpose. When this tank is about full (the product of half a week's run) the oil is pumped into the refining tank *B*. It will be observed that the delivery pipe is tapped into the tank near the bottom. This arrangement is for the purpose of avoiding the churning and excessive foaming contingent upon introducing the oil over the top of the tank, as is usually done. The oil is here refined, as previously described, and then allowed to stand, usually overnight, to allow the soapstock to settle out. The supernatant clear oil is then drawn off by means of a swivel arm and pumped to tank *C*, while the soapstock in the cone-shaped bottom is softened by steam coils and allowed to flow into the soapstock tank *d*, for subsequent filling into barrels. In a large refinery this is not the cheapest disposition, but in a small one it is probably the most practicable.

The small tank *a*, provided with barrel rests, receives the caustic soda for refining. The lye is transported by the monte-jus *a′* (closed tank provided with air pressure) to the weak-lye tank *b*, where the volume (or weight, if the tank is on scales) is adjusted for the given batch of oil. Tank *c* is for miscellaneous chemicals used from time to time for special treatment of the oil, such as salt and soda ash.

If the oil is to be washed, it is heated to approximately 165° F. and washed by spraying with about 5 per cent of hot water, or, better, with a 10 per cent brine solution, after which the water is allowed to settle out. The coils not only bring the oil up to the required temperature but assist in the separation of the moisture suspended in the oil. The water is drawn off from the bottom of the tank and flows into the general sewer, whereby any entrained oil may be recovered. If more than traces of moisture remain, the oil must be agitated and heated in order to dry it. The fuller's earth, previously weighed out in room *e* on the mezzanine floor over the tanks, is dropped into the oil in the tank *C*, which is covered during this operation to prevent the spreading of the dust. After agitating for about half an hour, the batch is pumped through the filter press *E* and allowed to run back into tank *C* by the sliding funnel until the cloths of the press have been covered and the oil flows clear. It is then run into tank *F*, which serves as a storage tank for the deodorizer.

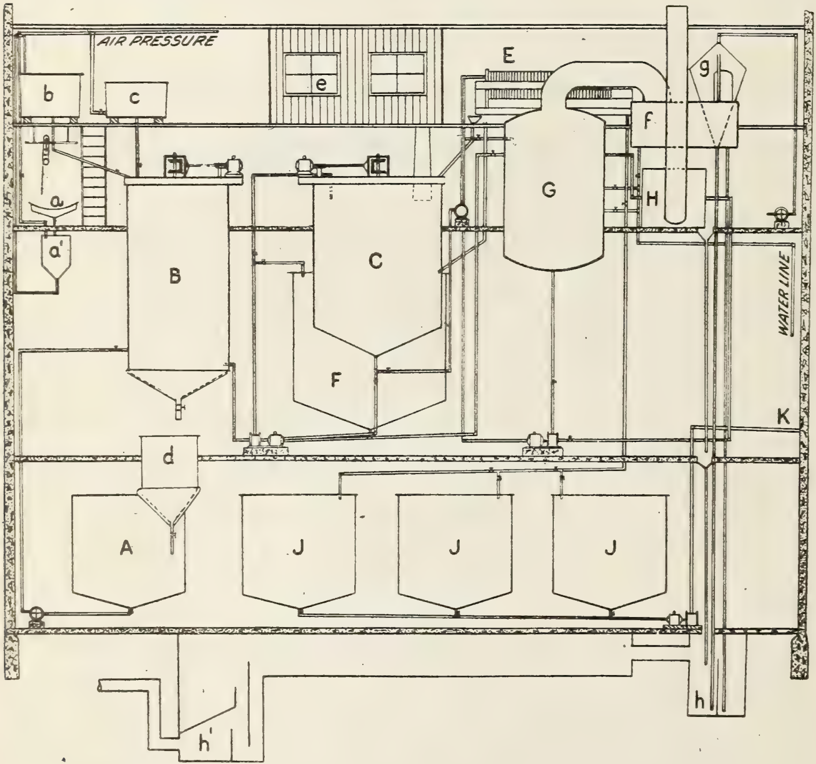

FIG. 7.—Plan of an equipment designed for refining 25,000 pounds of corn oil at one time.

Up to this point each unit of the equipment has been large enough to handle a batch of about 25,000 pounds of oil. The deodorizer, however, is designed for only half of this quantity. In the first place the larger the batch of oil the longer it must be subjected to the deodorizing treatment. This treatment benefits an oil up to a certain point, but if it is continued beyond that point the oil deteriorates. The smaller installation also means a lower overhead in the nature of certain fixed charges. The distribution of labor among the several operations in a refinery like the one under consideration is such that the two workmen have ample time to deodorize the oil in two batches.

The principle involved in deodorization is that of blowing live steam through the oil for several hours at temperatures above 400° F. under a very high vacuum. For the most effective results the steam must be brought into very intimate contact with the oil, which is accomplished by introducing the steam into the deodorizer through numerous minute perforations in a series of pipes emanating from a header.

In the industrial application of this principle several different procedures are followed. In some plants the oil is heated by closed steam coils to as high a temperature as is possible with the regular factory boiler steam and is then blown, either with some of the same steam or with a separate line of superheated steam. Others heat the oil with ordinary factory steam or with superheated steam in closed coils until nearly the required temperature is reached and then turn in the open steam, likewise superheated. The tendency seems to be to use open steam at as low a pressure as is practicable.

The great difficulty in heating the oil by superheated steam is that it is practically impossible to attain the desired temperature by such means. Since it is entirely feasible to carry superheated steam at a temperature of 600° F. it would appear to be a simple matter to use it to maintain oil at a temperature of 400° to 440° F. As a matter of fact, however, it is almost impossible to accomplish this. Consequently, the oil is often heated in a direct-fired furnace, designed very much like a direct-fired ordinary steam superheater, substituting, however, the oil stream for that of the steam. Any desired temperature can be attained by such an arrangement, the control is good, and the performance satisfactory.

It has been calculated that the consumption of coal in a direct-heat deodorizer is about one-fifth of that used in one heated by superheated steam, which, in a batch of the above size (25,000 pounds), amounts to more than one-third of a cent per pound of oil. Of course if other uses can be found for the heat of the steam after it leaves the coils of the deodorizer there must be a credit entered against the coal charged to deodorization. But in a small refinery the intermittent character of this additional use precludes any serious attempt at its utilization. Expedition and skill are required in handling such direct-fired equipment, and great care must be exercised or a batch of oil will be burned. The pumping capacity for forcing the oil through the heater must be ample in order to prevent scorching, and there must be protection against mishap, such as stoppage of flow, since this would result in ruining the entire batch.

In a large refinery it may be more practicable to locate the oil heater near the boiler room of the plant, so that one crew of firemen and the coal and ash handling equipment can take care of all the fires;

but in the case of a small refinery, such as is here considered (fig. 7), this may be impracticable. Accordingly, the heater *H* is located near the deodorizer *G* and is arranged to burn fuel oil. This not only obviates the cost of handling coal and ashes but affords ready control, a fairly constant temperature, and can be so regulated that it does not require the undivided attention of the operator. The two workmen of the refinery, previously referred to, should be able to handle this equipment. A very decided advantage in using oil instead of coal for fuel lies in the ease with which the fire can be extinguished in case the pumps stop. If coal costs $6 delivered and fuel oil $2.50 a barrel,[10] the cost for heating is about the same, but the oil has the advantage of better control, greater cleanliness, absence of charges for handling ashes, and the saving in fireman's wages.

The steam, with its charge of volatile odorous vapors, passes into the tank *f*, which functions both as a trap and as a preliminary condenser, and then passes into the jet condenser *g*, where a curtain of water entering near the top condenses the steam and discharges it into the hot well *h*. The condenser discharge pipe is longer than that required to produce a Torricellian vacuum and therefore insures a good working vacuum. However, certain noncondensible gases accumulate in the peak of the condenser *g*, operating to lower the vacuum. These gases are removed by the auxiliary dry-vacuum pump through the line entering the condenser at the top. The water for the condenser *g* is the same as that used in the preliminary condenser *f*.

After deodorization, the oil must be cooled to atmospheric temperature under a high vacuum; otherwise the deodorized oil will deteriorate by oxidation. In some plants the oil is cooled by running it through a series of jacketed pipes. In the plant here considered the cooling is effected by turning a stream of water into the closed steam coils in the deodorizer and circulating the oil by the regular deodorizing pump. Sometimes a fine precipitate is found in the oil, particles are taken up from the equipment, or other circumstances develop which make it necessary to filter the oil through a clean filter press before storing in tanks *J*. The oil is pumped from these tanks through pipe *K* into cars.

All floors drain into the hot well *h*, which overflows into *h'*. This serves as a trap for any oil which may be spilled and that which is carried over with the deodorization vapors.

It must be recognized that although this treatment will give a high-grade edible oil, it can not be expected that such oil can be used for salad purposes without further treatment. Most vegetable oils contain more or less solid glycerids, which settle out when the oil is chilled. In order, therefore, to be sure that an oil will remain clear over the rather wide range of temperatures obtaining in the economy of the ordinary household, the oil must be chilled below the lowest temperatures to which it is likely to be subjected and then filtered.

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[10] This price is about an average of fuel-oil prices for 1919. Prices for 1920 are not included because of the abnormal conditions which prevailed in the oil market during that year.

SUMMARY.

The process of preparing an edible oil from crude corn oil consists of three distinct operations: (1) Neutralization with caustic; (2) bleaching with fuller's earth; and (3) deodorizing with steam.

Two methods of refining average corn oil are found to be about equally satisfactory. The soapstock obtained in refining corn oil is soft and slimy, and special means are necessary to harden it. In one of the proposed methods a large excess quantity of caustic is used, while in the other the same result is obtained by adding soda ash after the break. A maximum temperature of 55° C. is recommended in the refining of corn oil.

The value of the chemicals used and the oil lost in the treatment is 0.628 cent per pound of neutralized oil with method 1 and 0.564 cent with method 2, of which more than 80 per cent is represented by the loss of oil.

The oil is bleached with fuller's earth according to the method generally used. Not less than 5 per cent of earth must be used. Corn oil does not bleach as much by this treatment as some of the other vegetable oils. The cost of the materials used and the oil lost in this bleaching process is about 0.22 cent per pound of bleached oil, of which approximately half represents the value of the oil lost.

The oil is deodorized by being blown with steam for several hours at temperatures above 400° F. under reduced pressure.

The general arrangement of a refinery equipped to handle two batches of 25,000 pounds of oil a week is described and the passage of the oil through the several processes is discussed.

The cost of refining corn oil in such a plant as here described is found to be approximately 1.6 cents a pound. This cost figure was obtained by determining as nearly as possible the several charges for chemicals, oil losses, fuel, labor, and overhead. The overhead includes interest on the equipment and depreciation on both equipment and building. The value of the equipment, exclusive of the building, is estimated at $40,000.

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